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Rubber Research Scheme (Ceylon).

RUBBER RESEARCH SCHEME CEYLON

Second Quarterly Circular

for

1927.

Reprinted from the *Tropical Agriculturist.*

July, 1927.

H. W. CAVE & CO., PRINTERS.

Explanatory Notes on Vulcanization Testing of Rubber.

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Report from the Imperial Institute

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Explanatory Notes on Vulcanisation Testing of Rubber.

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ESTATE Superintendents and others have frequently asked for a simple explanation of the meaning of figures given in reports dealing with the vulcanising properties of rubber samples. It is thought that the following notes may increase the value of such reports to non-technical readers.

With the exception of crepe soles and one or two minor products rubber is always used in the vulcanised form, and it has not been found possible by examination of raw rubber to predict what its properties will be after vulcanisation. Consequently in order to test the quality of a sample of rubber, it is necessary to vulcanise it under suitable conditions and then submit it to certain tests. Of recent years the question of " plasticity " of raw rubber has become of increased importance. This property is judged before vulcanisation, but can conveniently be included under the heading of vulcanisation tests.

From the point of view of the layman it is fairly accurate to say that " vulcanisation " or " curing " consists of causing rubber to combine with a certain proportion of sulphur. Under certain conditions this can be done without heat, being known as " cold vulcanisation," but more usually it is effected by heating a mixture of rubber and sulphur at a temperature of 140-150°C. If certain substances known as " accelerators " are added to the rubber the process takes place more rapidly, or alternatively can be carried out at a lower temperature.

For commercial purposes, rubber before vulcanisation is almost invariably " compounded " with various ingredients in addition to sulphur, which have the effect of modifying the properties of the vulcanised rubber to make it suitable for various uses. For testing purposes it was usual until recently to make the tests on rubber vulcanised with sulphur only, as this brings out differences between various samples of rubber. Since the introduction of organic accelerators it has been found advisable to make comparative tests of samples when vulcanised with sulphur only, and when vulcanised in presence of an accelerator. Certain accelerators only function in the presence of compounding ingredients so it is necessary for a suitable substance (usually

zinc oxide) to be added to the accelerator mix. Thus in a typical report from the Imperial Institute (see table I.) we find the tests given under two headings.

A. **Mixing** 90 Rubber, 10 Sulphur,

B **Accelerator Mixing** 90 Rubber, 5 Sulphur, 90 Zinc oxide, I Hexamine (accelerator).

Mastication Mixing.

Preparation of the raw rubber for vulcanisation consists of rolling through heated differentially geared rollers until the rubber becomes sufficiently soft and plastic to mix in the sulphur, etc. This process is known as " mastication." The sulphur and any other compounding ingredients are then added gradually and rolling is continued until they are thoroughly mixed with the rubber. Samples of plantation rubber vary in the amount of working required to soften the rubber, or in other words they vary in plasticity. This variability causes great inconvenience in manufacturing process. For many purposes such as manufacture of tubing, solid tyres, etc., the rubber after being mixed is " extruded " or forced through dies of the required shape, and if insufficiently plastic it emerges from the machine with a rough and uneven surface instead of being smooth and even. It has then to be sent back and reworked.

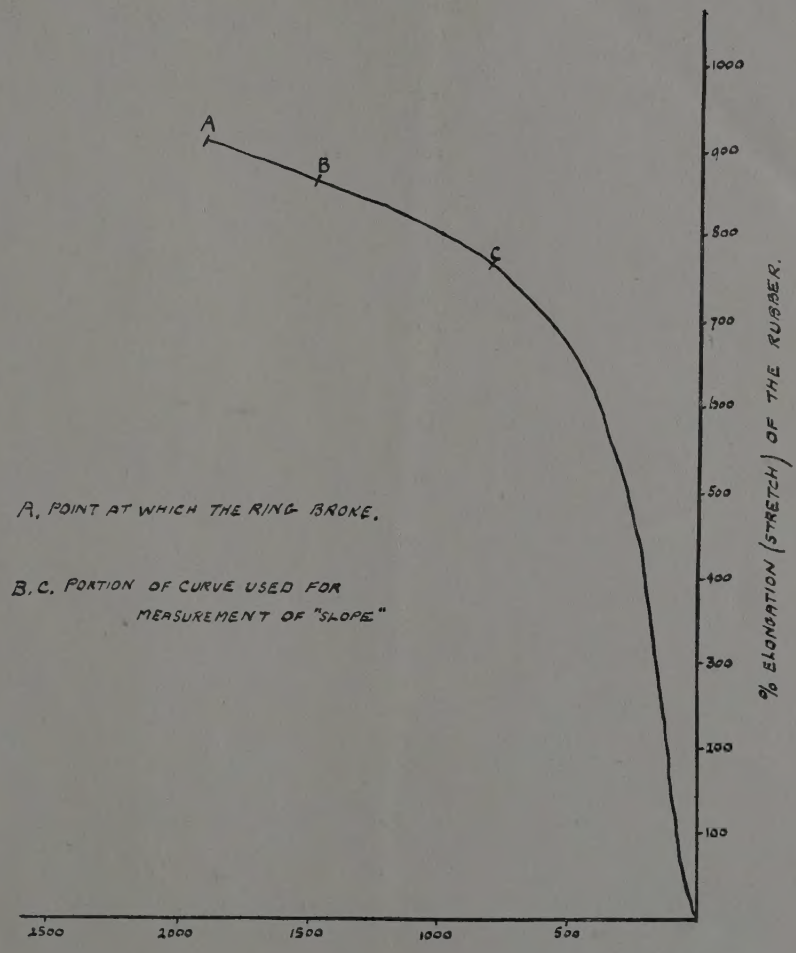
Plasticity Tests.

Two methods of measuring plasticity are in use at the Imperial Institute. In the first case a small piece of rubber of known size is heated to 100° C. and submitted to a known pressure for 30 minutes. The thickness of the sample after this treatment is a measure of the plasticity. Referring to Table 2 it will be seen that the figure for this test is given under the heading " D 30 " (D 30 can be taken to mean diameter after 30 minutes.) In this test the lower the figure obtained, the more plastic the rubber. It will be noticed that this test is made on the raw rubber and also after masticating and finally after mixing. Another test of plasticity which is now adopted, and which is much more sensitive than the foregoing, consists of extruding or forcing the rubber through a hole of given size under given pressure. The time taken to extrude a given weight is a measure of the plasticity of the rubber. In the table referred to, figures for this test are given under the heading "E.t." (E.t. can be taken to mean time of extrusion). This test is made after mastication and after mixing. In this case again a low figure represents rubber of good plasticity.

The amount of power consumed in masticating and mixing the rubber is also to some extent a measure of plasticity and in preparing the samples for plasticity tests mastication is carried out on rollers driven by an electric motor of which the power

DIAGRAM 1.

GRAPH DRAWN BY "SCHOPPER" TESTING MACHINE.



A. POINT AT WHICH THE RING BROKE.

B.C. PORTION OF CURVE USED FOR MEASUREMENT OF "SLOPE"

consumption can be measured. Rolling is continued until a certain fixed amount of current has been consumed. In Table 2, under the appropriate heading, is given the number of minutes required by each sample to consume the fixed amount of power. A high figure under this heading represents an easily workable rubber.

Time of Vulcanisation.

To turn from plasticity to vulcanisation tests, the first factor on which information is required is the rate of vulcanisation of the sample, *i.e.*, the length of time which the rubber must be heated with a fixed amount of sulphur at a fixed temperature in order to develop its maximum strength. Variability in rate of vulcanisation is a feature of plantation rubber and previously caused great inconvenience to the manufacturer, but since the introduction of organic accelerators it has lost some of its significance as suitable accelerators tend to equalise the rate of vulcanisation of different samples.

In Table I. it will be noted that there are two headings " standard cure " and " tensile optimum cure." Before explaining the meaning of these terms it is necessary to proceed to the next stage and describe the way in which the rubber is tested after vulcanisation.

Tensile Strength.

When vulcanisation is completed the sheets of rubber are allowed to stand for 24 hours, after which ring test pieces of accurately measured diameter and thickness are cut by means of a machine. The ring is placed in a machine known as the "Schopper" testing machine, and is stretched by applying a gradually increasing load. Stretching is continued until the ring breaks and the machine records the load required to break it, and also the amount which the rubber stretched before breaking. The thickness of the ring used for the test is only about $\frac{1}{4}$ inch but by calculation the breaking load or " tensile strength " per sq. inch is calculated. The amount of stretch is recorded under the heading " Elongation (per cent.) at break." While the ring is being stretched the Schopper machine automatically draws a graph representing the amount that the rubber stretches as the load is gradually increased, and a typical curve is shown in diagram I. The end of the curve represents the point at which the ring broke.

Under-vulcanised rubber stretches very easily. As vulcanisation proceeds, so the rubber becomes tougher, *i.e.*, to stretch it to a given extent a greater load is required. When over-vulcanised it becomes brittle. Vulcanisation to " standard cure " (in a rubber-sulphur mixing) is defined as vulcanising to a stage at which a load of 1,500 lb. per sq. inch (1·04 kg. per sq. mm) is required to stretch the rubber to 775 per cent. of its original length. " Tensile optimum cure," as the name implies,

consists of vulcanising to a stage at which the sample gives the highest tensile strength. For most purposes of comparison standard cure gives all the information that is required, and entails less work than the tensile optimum cure.

In general the tensile strength of a correctly vulcanised sample of plantation rubber will exceed 2,000 lb. per sq. inch. When a low figure for tensile strength is given in a report it must be studied in connection with the figure for "Elongation (stretch) at standard load." If this figure is higher than 775 per cent. it implies that the sample is under-vulcanised, and it is then known that if the time of vucanisation had been increased, improved figures for tensile strength would result. For example in Table 3 a result is given for a sample in which the tensile strength is 1,570 lb. per square inch which is a very low figure. The figure for " elongation at standard load " however is very high—943 per cent., showing that the sample is under-vulcanised and would have developed better tensile strength if vulcanised for a longer period. In this case it was not necessary to repeat the vulcanisation as the two samples under comparison were vulcanised to approximately the same extent (stretch at standard load 943 per cent. and 957 per cent. respectively) and could thus fairly be compared.

Slope.

Another heading given in reports is "slope" (see Table 3). Actually "slope" refers to the slope of the Schopper machine curve at a certain stage of stretching, shown in diagram I. It has been shown by Investigators that the slope of the curve at this stage of the test is to some extent an index of the quality of the rubber. A low figure for slope indicates rubber of good quality and "slope" figures for good quality plantation rubber in a rubber-sulphur mix are usually not higher than 40. In an accelerator mixing (Table I.) the "slope" figure has a different value

Ageing Tests.

Another heading in reports is " Ageing Tests." It is common knowledge to every user that rubber gradually perishes, particularly when exposed to light and air, and it will be appreciated that it is of importance to compare the properties of different samples of rubber in this respect. Perishing of rubber takes place slowly and it would be a great handicap if the Investigator had to wait 12 or 18 months to observe the effects of keeping the rubber, in addition to the fact that conditions of storage would have a considerable influence on the result. It has been found that if the sample is heated in an oven at 70° C. in a current of air, a change in tensile strength takes place similar to that which occurs during ordinary storage or use, and this is known as " artificial ageing." Heating in the oven for 24 hours has

approximately the same effect as 6 months natural ageing. From Table 4 it will be seen that tests are made after 48, 96, and 144 hours' artificial ageing. An average sample of plantation rubber begins to show some deterioration in tensile strength after 96 hours' ageing. It will be noted in this case that the samples actually showed improvement of strength after 48 hours' ageing and it is necessary to explain that for ageing tests the samples are purposely somewhat under-vulcanised. This corresponds with commercial practice, as full vulcanisation to the standard used for physical testing would give a product with impaired ageing properties.

It is hoped that the above notes will be of assistance in explaining in a general way the meaning of the figures given in reports of vulcanisation tests. In certain respects the explanations are necessarily incomplete, as full explanation would involve technical discussion which would not be of interest to the reader for whom these notes are intended.

Table I.

(Extract from "First report on rubber prepared with sodium silico fluoride as a coagulant." R.R.S. 4th Quarterly Circular for 1924, p. 8).

A. Mixing 90 Rubber, 10 Sulphur.

	Time of cure (mins.)	Tensile strength (lbs./sq. in)	*Elongation.* At Break, per cent.	*Elongation.* At standard load per cent.	Slope.
Standard Cure (Elongation at load of 1·04 kgs sq. mm. = 775 per cent.)					
Present sample ..	150	2,390	874	784	39
(Smoked Sheet average of 5 recent samples) ..	107	2,440	867	777	40
Tensile Optimum Cure (Maximum tensile strength developed 72 hours after vulcanisation.) ...					
Present sample ..	150	2,390	—	784	—
Smoked Sheet (average of 5 recent samples) ...	113	2,510	—	754	—

B. Accelerator Mixing.

90 Rubber, 5 Sulphur, 90 Zinc oxide, 1 Hexamine.

	Time of Cure (mins)	Tensile strength (lbs./sq. in)	*Elongation.* At Break per cent.	At standard load per cent.	Slope.
Standard Cure (Elongation at load of 1·04 kgs/sq. mm. = 480 per cent.)					
Present sample ...	60	2 850	633	475	46
Smoked Sheet (average of 5 recent samples ...	37	2,420	604	481	47
Tensile Optimum Cure (Maximum tensile strength developed 72 hours after vulcanisation).					
Present Sample ...	50	2,960	—	495	—
Smoked Sheet (average of 5 recent samples.) ...	45	2,760	—	458	—

Table II.

(Extract from "Report on the effect of para-nitrophenol on the plasticity, vulcanising, and ageing properties of blanket crepe." R.R.S. 4th Quarterly Circular for 1926, p. 11.)

(I). Plasticity Tests.

Sample No.	Form of Rubber	Time of mastication for power consumption of 450 watt hours	Time of mixing for power consumption of 150 watt hours	Plasticity		
				Raw Rubber	Masticated Rubber	Rubber sulphur Mixing (90·10)
		(mins)	(mins)	D 30	D30. E t	D30 E.t.
1261	Blanket crepe (control) ...	23½	11	167	80 70 ·8	72 43 ·6
1262	Blanket crepe soaked in 0·1 per cent. para-nitrophenol for 30 minutes ...	23½	11	171	86 72 ·5	73 46 ·6
1263	Blanket crepe from latex coagulated with acetic acid and 0·1 per cent. para-nitrophenol (calculated on dry rubber) ...	22½	10½	174	87 75 ·3	72 46 ·5

D 30 = Thickness (in hundredths of a millimetre) of sphere 0·4 gram in weight after pressing in plasticity press at 100°C for 30 minutes.

E. t. = Time (in minutes) required to extrude fixed volume at 90°C

Table III.

(Extract from "Third report on rubber prepared with sodium silico fluoride as a coagulant." R.R.S. 4th Quarterly Circular for 1924, p. 12).

Rubber Sulphur Mixing: 90: 10.

Coagulant	Time of cure (mins)	when tested	Tensile strength (lb. sq. in)	Elongation		
				At Break (per cent.)	At standard load (per cent.)	slope
Sodium-silico fluoride	92	Before ageing	1,570	953	943	40
		After ageing	1,930*	931	877	39
Acetic acid (control)	80	Before ageing	1,480	960	957	41
		After ageing	2,070*	957	893	40

* By heating for 20 hours at 70°C. in a current of air.

Table IV.

(Extract from "Report on the effect of para-nitrophenol on the plasticity, vulcanising, and ageing properties of blanket crepe." R.R.S. 4th Quarterly Circular for 1926, p. 12).

Vulcanising and Ageing Tests.

Sample No.	Form of Rubber	Time of vulcanisation.	Period of ageing at 70°C	Tensile strength	Elongation at load of 1·04 kgs./sq mm.
		(mins)	(hrs)	(lbs./sq. in)	(per cent.)
1261	Blanket crepe (control)	120	nil	1780	890
			48	2240	793
			96	1880	749
			144	280	—
1262	Blanket crepe soaked in 0·1 per cent. para-nitrophenol for 30 minutes.	120	nil	1910	887
			48	2080	786
			96	1790	744
			144	340	—
1263	Blanket crepe from latex coagulated with acetic acid and 0·1 per cent. para-nitrophenol (calculated on dry rubber)	120	nil	2120	860
			48	2390	768
			96	1680	726
			144	330	—

The Effect on Yield of Manuring Rubber with Nitrate of Soda.

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Mycologist, Rubber Research Scheme, Ceylon.

THE importance of nitrogen in the life of plants has for many years been well recognised, and as a constituent of manures its effect has been closely studied. It has been found that this effect is mainly upon the green parts of the plant, particularly the foliage, which in the majority of cases becomes more luxuriant and darker in colour under applications of nitrogen in an available form to the roots. The effects are most easily observed when the nitrogen is supplied alone in a chemical which contains no other substance of important nutritive value to the plant. Such a material is provided by nitrate of soda or "chili saltpetre."

In an attempt to determine the effect, if any, of nitrate of soda applied as a manure to rubber an experiment was carried out on an estate in the Kalutara District in collaboration with the Mycologist of the Rubber Research Scheme. The points to be investigated were primarily the effect on the incidence of secondary leaf-fall and secondarily on the yield. The effect on secondary leaf-fall was reported in the 3rd Quarterly Circular for 1925. The present report is concerned with the effect on yield.

Description of Experiment.—A plot was chosen containing some seventeen hundred trees planted in well defined rows of about thirty trees each. This area was divided into ten two-acre blocks numbered from the beginning 1B, 1A, 2B, 2A, 3B, 3A, 4B 4A, 5B, 5A. The whole area includes a valley, so that the field slopes gently down from one end (plots 5A and B) to the other while the individual blocks slope steeply on each side of a central stream. The plots were treated as follws:—

1B. Control, untreated.
1A. Manured, nitrate of soda 5 cwt. per acre.
2B. Manured, nitrate of soda 5 cwt. per acre.
2A. Control, plain forked.
3B. Control, plain forked.
3A. Manured, nitrate of soda 5 cwt. per acre.
4B. Manured, nitrate of soda 5 cwt. per acre.
4A. Control, untreated.
5B. Control, untreated.
5A. Manured, nitrate of soda 5 cwt. per acre.

The manure was applied in two doses of $2\frac{1}{2}$ cwt. per acre in March, 1923, March, 1924, and again in February-March, 1925. The ground was " enveloped forked " and the manure spread in the furrows. Prior to the experiment the area was last manured in 1920.

All trees were tapped on a $\frac{1}{2}$ circumference on alternate days, and yield figures for each block in pounds of dry rubber per tapping were recorded from the beginning of the experiment.

In the examination which follows the yields for each set of blocks of the same kind are considered together and the average yield per one hundred trees per tapping calculated. (This is taken as the basis, as it gives figures of an order convenient to deal with.)

It should be pointed out that it is necessary to consider all the plots of one kind together because no reliance can be placed on figures obtained from single, unduplicated plots or by the comparison of the plots in pairs. Too many factors other than the one under consideration may be concerned in producing any difference there may be between any pair of single plots. The existence of only two forked and three unforked control plots tends to impair the values of the figures.

The Total Yields for the three years are as follows:—

Manured Blocks.

	Total Yield in lbs.	No. of trees.	No. of tappings.	Yield per 100 trees per tapping.
1A.	2580·5	195	363	3·646
2B.	2644·6	183	362	3·991
3A.	2473·5	178	349	3·982
4B.	2490·7	176	363	3·898
5A.	1963·4	175	354	3·170
				5/18·687
				3·737 lb.

Forked Control Blocks.

	Total Yield in lbs.	No. of trees.	No. of tappings.	Yield per 100 trees per tapping.
2A.	2319·6	162	349	4·103
3B.	2280·9	175	356	3·662
				2/7·765
				3·888 lb.

Unforked Control Blocks.

1B.	2201·4	191	369	3·124
4A.	2065·7	178	348	3·335
5B.	1518·5	164	352	2·631
				3/9·090
				3·030 lb.

Comparing the figures in the last column we have the following results:—

Manured Blocks Compared with Unforked Controls.

Difference in mean yield per 100 trees per tapping.

$= 3·737 - 3·030$
$= 0·707$
$= 23·3\%$ increase on controls.

Manured Blocks Compared with Forked Controls.

Difference in mean yield per 100 trees per tapping.

$= 3·737 - 3·883$
$= 0·146$
$= 3·76\%$

i.e., there is a *decrease* of 3·76 per cent. for the manured as compared with the control blocks.

Forked Controls Compared with Unforked Controls.

Difference in mean yield per 100 trees per tapping.

$= 3·883 - 3·030$
$= 0·853$
$= 28·15\%$ increase

It is apparent that the only real difference in yield is between the forked and unforked plots, for the manured plots (which are, of course, forked) while showing a considerably higher yield than the unforked controls, are rather *lower* than the average of the forked controls.

It cannot therefore be said from this experiment that nitrate of soda applied as a manure results in any definite increase in yield through its own direct action. There is, however, some evidence that the operation of forking has some stimulating effect reflected in an increase in yield.

Possible Contamination of Latex with Copper Compounds as a Result of Spraying the Trees with Bordeaux Mixture.

Report from the Imperial Institute.

SUBSTANCES containing copper are known to have a harmful effect upon rubber, and recent experiments carried out by the Ceylon Rubber Research Scheme have demonstrated that Bordeaux mixture, if introduced into latex, causes the rubber to become soft and plastic on keeping and to perish quickly when vulcanised (Fourth Quarterly Circular, 1926, p. 13). Even a trace of Bordeaux mixture in the latex has an appreciable effect upon the keeping properties of both the raw and vulcanised rubber.

In these circumstances it was considered desirable in Ceylon to determine whether spraying the crown of the trees with Bordeaux mixture in the usual way is likely to introduce copper into the rubber.

Two series of experiments were carried out to determine this point. In the first no rain fell between spraying and tapping, and the ageing properties of the rubber prepared from latex collected 1 day after spraying were as good as those of a control sample prepared before spraying, showing that no copper from the Bordeaux mixture had entered the latex.

In the second series of experiments, a certain amount of rain fell after spraying and the ageing properties of the rubber prepared 1 and 3 days afterwards were not quite as good as those of the control sample prepared before spraying. The difference between the samples however was not sufficiently marked to suggest the presence of copper compounds. Previous experiments have shown that the smallest amount of copper which can be detected by chemical means has an appreciable effect on the ageing qualities.

The results of the tests, details of which are given below, show that in these experiments spraying the trees with Bordeaux mixture did not introduce sufficient copper into the latex to cause deterioration of the rubber on keeping. It cannot be too strongly emphasised, however, that great care should be exercised in preparing rubber after spraying operations, as the presence of even traces of copper in rubber is detrimental. While the rubber is in the raw state the effect may not become obvious for many months but nevertheless all articles manufactured from such rubber will quickly perish.

Details of Experiments and Results of Tests.

Experiment No. 1.—A control sample of crepe (No. 1269) was prepared from the bulked latex from a group of 100 trees. The trees were sprayed with normal Bordeaux mixture the next day (3 gallons per tree) and a second sample of crepe (No. 1270) was prepared the day after spraying.

Experiment No. 2.—A control sample of crepe (No. 1271) was prepared from a group of 100 trees three days before spraying. The next sample (No. 1272) was prepared the day after spraying; in the interval between spraying and tapping 0·1 inch rain fell. During the next two days 1·1 inch rain fell after which a third sample (No. 1273) was prepared.

On arrival at the Imperial Institute all the samples appeared to be of good quality.

The samples were submitted to plasticity, vulcanising and ageing tests six months after preparation.

The results of the plasticity tests are as follows:—

Table I.

Experiment	Sample No.	Particulars	Time of mastication for power consumption of 450 watt hours	Time of mixing for power consumption of 150 watt hours	Raw Rbber	Masticated Rubber		Mixed Rubber	
					D_{30}*	D_{30} *	Et †	D_{30}*	Et †
			(mins)	(mins)					
1	1269	Control	25½	10½	146	68	35·1	59	24·1
	1270	Prepared 1 day after spraying	25	10½	143	74	41·4	64	31·2
2	1271	Control	24	11	150	70	49·7	63	34·1
	1272	Prepared 1 day after spraying during which 0·1 inch rain fell	26	11	117	69	33·3	60	24·0
	1273	Prepared 3 days after spraying during which 1·2 inch rain fell	26	11	141	66	41·8	61	24·0

*D_{30} = Thickness (in hundredths of a millimetre) of sphere 0·4 grasof in weight after pressing in plasticity press at 100°C mr 30 minutes.

† Et = Time in minutes required to extrude fixed volume at 90°C.

All the samples, including the controls, are more plastic than the average figures for estate crepe.

In the first experiment, in which no rain fell between spraying and tapping, the crepe prepared after spraying was less plastic than the control. In the second experiment the two samples prepared after spraying are more plastic than the control and it is possible that the rain washed traces of copper on to the tapping surface. The differences in plasticity however are only small.

The following are the results of vulcanising and ageing tests:—

Table II.

Experiment	Sample No.	Particulars	Time of vulcanisation.	Period of ageing at 70°C.	Tensile Strength	Elongation at load of 1·04 kgs./sq. mm.
			(mins.)	(hrs.)	(lbs./sq. in.)	(per cent)
1	1269	Control	120	nil	2130	855
				48	2230	747
				96	1780	712
				144	290	—
	1270	Prepared 1 day after spraying	124	nil	1800	867
				48	2300	750
				96	1900	710
				144	200	—
2	1271	Control	124	nil	2110	853
				48	2200	760
				96	2040	707
				144	290	—
	1272	Prepared 1 day after spraying during which 0·1 inch rain fell	135	nil	1920	840
				48	2300	727
				96	1520	676
				144	260	—
	1273	Prepared 3 days after spraying during which 1·2 inch rain fell	125	nil	2070	841
				48	2330	756
				96	1670	676
				144	280	—

There is no important difference between the vulcanising and ageing properties of any of the samples. Samples 1272 and 1273 are slightly inferior to the control (1271), again suggesting that a trace of copper may have been washed by the rain on to the tapping surface. The difference between the results given by the three samples (1271-1273) however is so small as to be of no practical importance.

IMPERIAL INSTITUTE, LONDON,
March 23rd, 1927.

Note added in Ceylon.

The samples referred to above were prepared in connection with a spraying experiment which was carried out under scientific supervision. It was considered desirable by the Technical Committee of the Rubber Research Scheme, that these results should be amplified by tests on rubber from areas sprayed under routine estate conditions. A South Indian Estate has kindly supplied samples for the purpose and these are being forwarded to the Imperial Institute for examination.

(Sgd.) T. E. H. O'BRIEN,
Chemist,
Rubber Research Scheme.

NOTICES.

SUBSCRIPTIONS.

Arrangements have now been made for Bulletins of the Ceylon Rubber Research Scheme to be made available to non-contributors to the Scheme at the rate of Rs. 15·00 per annum, post free.

OFF-COLOURED CREPE.

The Chemist to the Rubber Research Scheme would be glad to get into touch with the Superintendent of any estate who is troubled with off-colour crepe, and who thinks that this may be due to impurities in the water used for manufacture.

"HYDROLEX".

Glass Hydrometers for testing latex ("Hydrolex") as specified and as recommended by the Rubber Research Scheme (Ceylon) may be obtained at a cost of Rs. 12·50 each from :—

Messrs. WALKER, SONS & Co., Ltd.
Engineering & Estate Supplies Department.
Colombo.